



Chloramphenicol photo-Fenton degradation and toxicity changes in both surface water and a tertiary effluent from a municipal wastewater treatment plant at near-neutral conditions



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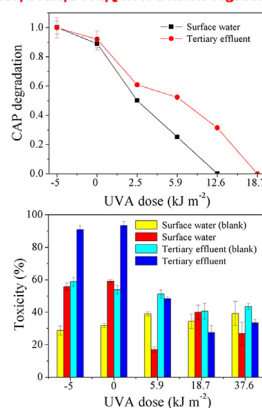
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HIGHLIGHTS

- The FeOx/photo-Fenton process can be applied at near-neutral conditions.
- CAP was efficiently degraded in both SW and TE.
- Organic load and $\text{CO}_3^{2-}/\text{HCO}_3^-$ ions reduced the kinetics of CAP degradation in TE.
- The best results were achieved under sunlight, with added savings in energy costs.
- The solar/photo-Fenton process promoted CAP degradation and a non-toxic effluent.

GRAPHICAL ABSTRACT

CAP/Solar/FeOx/photo-Fenton degradation



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ABSTRACT

Antibiotics are known to be refractory to natural biological degradation and conventional water treatments. Their presence in the effluent of sewage treatment plants and surface waters may harm aquatic environments, making it important to evaluate new technologies and to assess their degradation potential at near-neutral conditions. This study thus describes the degradation of the antibiotic chloramphenicol in both surface water and a tertiary effluent from a municipal wastewater treatment plant by the photo-Fenton process under black-light and solar irradiation conditions. The best results in surface water at an initial pH of 5.8 (natural of this matrix) were obtained using $48 \mu\text{mol L}^{-1}$ FeOx and $44 \mu\text{mol L}^{-1}$ H_2O_2 , with a $144 \mu\text{mol L}^{-1}$ oxalate added at 10 min, reaching 88% degradation after 15 min of black-light irradiation. However, in a previous step where the pH was adjusted from 7.7 to 6.0, high FeOx ($192 \mu\text{mol L}^{-1}$) and H_2O_2 ($1500 \mu\text{mol L}^{-1}$) concentrations were needed to achieve 79% chloramphenicol degradation in the tertiary effluent after 20 min of black-light irradiation. Using the same experimental conditions for each matrix, the best results, in terms of chloramphenicol degradation rate as by-products of low toxicity, were obtained under solar irradiation, as confirmed by the blank experiments using the matrices in the absence of chloramphenicol. These results show the viability and effectiveness of FeOx

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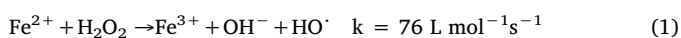
as a photocatalyst for the treatment of wastewaters containing this pollutant at near-neutral conditions under solar irradiation, as well as the added savings in energy costs associated with the photo-Fenton treatment.

1. Introduction

The presence of pharmaceuticals in different water matrices and specific antibiotics has been documented and reported as an emerging environmental issue [1]. The most important and continuous input into aquatic compartments is through municipal wastewater treatment plants, which are inefficient in the removal of these pollutants [2].

Chloramphenicol (CAP; Fig. 1-SM) is widely used for treating infections caused by Gram-positive and Gram-negative bacteria, as well as other groups of microorganisms [3]. It has been detected in surface waters (SW) at concentrations between 30 and 80 ng L⁻¹ in Kenya and 0.6–11.2 µg L⁻¹ in China [4,5]. Concentrations of 0.56 µg L⁻¹ in Germany and 47 µg L⁻¹ in China have also been reported in sewage treatment plant (STP) effluents [4,6]. Its presence in aquatic environments may thus pose detrimental effects on aquatic organisms [7,8].

As a consequence of the limitations of conventional wastewater treatment plants in removing antibiotics compounds, several efforts are being made to find out ways to improve wastewater treatments, such as advanced oxidation processes (AOPs), based on the formation of highly-reactive hydroxyl radicals (HO·). Among the AOPs, special attention has been focused on heterogeneous photocatalysis and photo-Fenton processes due the possibility of using sunlight as the radiation source [9]. However, the use of a photo-Fenton process (Eqs. (1) and (2)) has the advantage of being a homogeneous process, avoiding the removal of the catalyst when the iron concentration used is below the legislated value.



The main disadvantage of applying the photo-Fenton process in environmental matrices is that acidic media, with pH levels between 2.5 and 3.0, are needed to avoid iron precipitation [10]. The use of iron complexes is one possibility to solve this limitation, since they permit the process to work at near-neutral conditions [11,12].

The degradation of CAP by different AOPs, such as UV/H₂O₂ [13], ozonation [14], heterogeneous photocatalysis [15], Fenton [16,17], solar photoelectro-Fenton [18], and photo-Fenton [16,17,19], has been evaluated using ultrapure water, initial CAP concentrations in the mg L⁻¹ range, and a pH around 3 (the last being specific to Fenton degradation processes), all of which are far from the concentration ranges and conditions found in natural water and wastewater effluent. Therefore, this investigation presents a number of important advances in the photo-Fenton process for CAP degradation, such as:

- Evaluation of the degradation of this antibiotic in two complex and real media, SW and tertiary effluent (TE), since it has been found in environmental waters;
- Evaluation of the operational parameters and determination of the best conditions to degrade CAP at near-neutral conditions;
- Evaluation of the potential and effectiveness of the modified photo-Fenton process under black-light and solar irradiation as an alternative to the treatment of this kind of pollutant in SW and TE by monitoring the changes in acute toxicity to the bacteria *Vibrio fischeri*.

Considering that the best results of CAP degradation in distilled water by the photo-Fenton process at near-neutral conditions were obtained using the iron complex ferrioxalate, FeOx [20], this iron source will be used as the catalyst to evaluate the degradation of CAP in these two aqueous matrices (SW and TE). In addition, this oxalate

ligand is considered neither recalcitrant to biodegradation nor toxic to aquatic fauna [11].

2. Experimental

2.1. Reagents

All solutions were prepared using ultrapure water (18.2 MΩ cm) from a Milli-Q water purification system and analytical grade reagents. The CAP standard was purchased from Sigma-Aldrich, with a certified purity higher than 99 wt-% that was used as received. HPLC grade methanol (J. T. Baker) was used for the HPLC-DAD analyses. H₂SO₄ and NaOH (Vetec) were used for pH adjustment. Potassium ferrioxalate, (K₃Fe(C₂O₄)₃·3H₂O) – FeOx, prepared and purified as described in the literature [21], was used to prepare aqueous stock solutions containing 12 mmol L⁻¹ of iron. Other reagents employed in this study were: bovine catalase (Sigma-Aldrich), NH₂OH·HCl (Vetec), CH₃COONa (Pan-reac), and C₄K₂O₉Ti·2H₂O, H₂O₂ (30 wt-%), Na₂SO₃, 1,10-phenanthroline, and K₂C₂O₄·H₂O, all from Synth.

2.2. SW and TE sampling

SW and TE samples from a municipal wastewater treatment plant were collected during the autumn and spring 2017 to evaluate the application of the photo-Fenton process and its matrix effects on CAP degradation. The SW sample was collected directly from a river whose water, after conventional treatment, is supplied to the city of Uberlândia. The TE sample was collected after complete treatment from a wastewater treatment plant that conducts preliminary, anaerobic, and tertiary treatment (coagulation-flocculation-flotation). This plant serves about 95% of the population of Uberlândia, Brazil (18°55'08"S, 48°16'37"W), a city with more than 670,000 inhabitants. Once collected, the samples were kept under refrigeration for two to three weeks to conduct the degradation experiments. The main parameters determined for both samples are shown in Table 1.

2.3. Degradation

Due to the low CAP concentration determined from SW and the STP effluents (ranging from 0.3 to 80 µg L⁻¹) [4–6], the decision was made

Table 1
Main parameters determined from the SW and TE samples.

Parameter	SW	TE
Alkalinity as CaCO ₃ (mg L ⁻¹)	22 ^a	269 ^a
Conductivity (µS cm ⁻¹)	7.2 ^a	846 ^a
BOD ₅ (mg L ⁻¹)	3.7 ^b	95 ^b
COD (mg L ⁻¹)	13 ^a	202 ^a
pH	5.8 ^a	7.7 ^a
Total dissolved solids (mg L ⁻¹)	< 10 ^b	27 ^b
Turbidity (nephelometric units)	5.8 ^b	55 ^b
Chloride (mg L ⁻¹)	1.4 ^b	174 ^b
Dissolved iron (mg L ⁻¹)	0.5 ^a	0.5 ^a
Phosphorus (mg L ⁻¹)	< 0.05 ^b	0.9 ^b
Ammoniacal nitrogen (mg L ⁻¹)	< 0.2 ^b	32 ^b

BOD₅ = biochemical oxygen demand after five days.

COD = chemical oxygen demand.

^a Dissolved Fe without acid digestion.

^a Measured in our laboratory.

^b Data furnished by the Department of Water and Sewage in Uberlândia (DMAE).

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